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INTERNATIONAL PRELIMINARY EXAMINATION REPORT  
(PCT Article 36 and Rule 70)

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Applicant's or agent's file reference 9572WO		<b>FOR FURTHER ACTION:</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/PEA/416)	
International application No. PCT/NL 03/00921	International filing date (day/month/year) 22.12.2003	Priority date (day/month/year) 27.12.2002	
International Patent Classification (IPC) or both national classification and IPC C07C45/85			
Applicant DSM IP ASSETS B.V. et al.			
<p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 5 sheets, including this cover sheet.</p> <p><input checked="" type="checkbox"/> This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of 3 sheets.</p>			
<p>3. This report contains indications relating to the following items:</p> <p>I <input checked="" type="checkbox"/> Basis of the opinion</p> <p>II <input type="checkbox"/> Priority</p> <p>III <input type="checkbox"/> Non-establishment of opinion with regard to novelty, inventive step and industrial applicability.</p> <p>IV <input type="checkbox"/> Lack of unity of invention</p> <p>V <input checked="" type="checkbox"/> Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</p> <p>VI <input type="checkbox"/> Certain documents cited</p> <p>VII <input type="checkbox"/> Certain defects in the international application</p> <p>VIII <input type="checkbox"/> Certain observations on the international application</p>			
Date of submission of the demand  05.07.2004		Date of completion of this report  11.02.2005	
Name and mailing address of the international preliminary examining authority:   European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465		Authorized Officer  Romano-Götsch, R  Telephone No. +49 89 2399-8874  	

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. **PCT/NL 03/00921**

**I. Basis of the report**

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

**Description, Pages**

1-10 as originally filed

**Claims, Numbers**

1-16 filed with telefax on 22.12.2004

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).  
☐ the language of publication of the international application (under Rule 48.3(b)).  
☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.  
☐ filed together with the international application in computer readable form.  
☐ furnished subsequently to this Authority in written form.  
☐ furnished subsequently to this Authority in computer readable form.  
☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.  
☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:  
☐ the claims, Nos.:  
☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)).

*(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)*

6. Additional observations, if necessary:

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. . PCT/NL 03/00921

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability;  
citations and explanations supporting such statement**

**1. Statement**

Novelty (N)	Yes: Claims	1-17
	No: Claims	
Inventive step (IS)	Yes: Claims	1-17
	No: Claims	
Industrial applicability (IA)	Yes: Claims	1-17
	No: Claims	

**2. Citations and explanations**

**see separate sheet**

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/NL 03/00921

**Re Item V**

**Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

The following documents, cited in the search report, are referred to:

D1: GB-A-2 028 329 (STAMICARBON) (1980-03-05) ( cited in the application)

D2: US-A-3 668 256 (BRUNDEGE JASPER A)(1972-06-06)

D3: US-A-5 667 644 (MORI TOMOYUKI ET AL)(1997-09-16)

D4: GB-A-1 382 849 (BASF AG) (1975-02-05)

**Novelty**

The present application meets the requirements of Art. 33(2) PCT because the subject-matter of claims 1-17 is novel over the cited prior art.

D1, regarded as the closest prior art, is directed to a process for purifying a mixture comprising cyclohexanone and one or more aldehydes, comprising subjecting the cyclohexanone and aldehydes to an aldol condensation reaction in the presence of NaOH in a separate vessel; washing with water the so-obtained mixture to remove the excess NaOH and finally distill the washed organic mixture (p.1, lines 79-120).

The distillation is performed in two steps: a fraction with a lower boiling point than cyclohexanone (light fraction) is distilled off and discharged. The heavy fraction comprising the cyclohexanone is passed through a second distillation column; wherein cyclohexanone is distilled and recovered (see p.2 , lines 115-125).

The claimed matter on file differs from the disclosure of D1 in that (1) the aldol condensation, the washing step and the distillation occur in one distillation column and (2) less than 100 weight ppm water is present at the bottom of the distillation column.

D2 is directed to a process for purifying crude acetone containing aldehydes as impurities.

D3 and D4 are not relevant to the novelty or the inventive step of the present application.

D3 is directed to a process for the preparation of a mixture of cyclohexanone and cyclohexanol by oxidation comprising the oxidation by a gas containing molecular oxygen and treatment with an alkali metal hydroxide or carbonate. The so-obtained mixture is subjected to distillation to achieve a mixture of cyclohexanol and cyclohexanone.

D4 discloses a process for producing dimerized aldehydes while minimizing the formation of trimers, tetramers etc., i.e. to a completely different technical problem.

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/NL 03/00921

Inventive Step

Departing from D1, the problem to be solved by the application is the provision of an alternative process for reducing the aldehyde concentration in a mixture comprising cyclohexanone and one or more aldehydes.

The solution proposed in the application is a process comprising distilling the mixture of cyclohexanone and aldehydes in a distillation column in the presence of an alkaline compound, wherein in the bottom of the distillation column less than 100 weight ppm water is present.

D1 teaches removing the aldehydes from the crude cyclohexanone by aldol condensation in the presence of sodium hydroxide in one vessel, followed by a washing step and recovering the cyclohexanone by distilling the washed organic mixture.

The distillation is performed in two steps: a fraction with a lower boiling point than cyclohexanone (light fraction) is distilled off and discharged. The heavy fraction comprising the cyclohexanone (and the remaining water) is passed through a second distillation column, wherein cyclohexanone is distilled and recovered (see p.2, lines 115-125).

D2 teaches removing aldehydes in crude acetone by combination of the chemical treatment and distillation performed in a single multi-plate distillation tower.

D2 teaches removing aldehydic impurities from crude acetone in a distillation column using an alkaline agent and under conditions wherein a single acetone-rich phase is formed in the reaction zone of the column, and recovering pure acetone from the head of the column (column 1, line 53, -column 2, line 75). The acetone-rich liquid phase contacts the upflowing vapours of acetone and aldehydes, converting the aldehydes to nonvolatile polymers, which tend to settle in the bottom of the column with water and the spent alkali, and which are removed through an outlet. D2 does not suggest a distillative process wherein it is required less than 100 weight ppm water is present that the bottom of the distillation column.

In view of D1 alone or combined with D2, the skilled person would not have arrived to the process on file in a straightforward manner.

The subject matter of independent claim 1 and dependent claims 2-16 appears to meet the requirements of Art. 33(3) PCT.

9572WO

22 December 2004

Enclosure 1.1

AMENDED SET OF CLAIMS

1. Process for reducing the aldehyde concentration in a mixture comprising cyclohexanone and one or more aldehydes, said process comprising distilling  
5 said mixture in a distillation column in the presence of an alkaline compound, wherein in the bottom of the distillation column less than 100 weight ppm of water is present.
2. Process according to claim 1, wherein said mixture is a mixture comprising cyclohexanone, cyclohexanol, low boiling compounds and high boiling  
10 compounds and wherein said distilling involves separation of low boiling compounds to obtain a top product comprising low boiling compounds and a bottom product comprising cyclohexanone, cyclohexanol and high boiling compounds.
3. Process according to any one of claim 1 to 2, wherein the aldehydes are  
15 hexanal and/or pentanal.
4. Process according to claim 2 or 3, wherein the distillation is effected at a top temperature of between 45 and 130 °C and a bottom temperature of between 105 and 190 °C.
5. Process according to any one of claim 1 to 4, wherein the process comprises  
20 feeding a solution comprising water and the alkaline compound to the distillation column at a level above the bottom of the distillation column.
6. Process according to any one of claim 1 to 5, wherein the process comprises feeding the mixture to said distillation column and feeding a solution  
25 comprising water and the alkaline compound to the distillation column at a level above the bottom of the distillation column.
7. Process according to any one of claim 1 to 5, wherein the process comprises feeding the mixture to the distillation column at a level above the bottom of the distillation column and introducing a solution comprising water and the alkaline compound into the mixture prior to said feeding.
- 30 8. Process according to any one of claim 1 to 7, wherein the alkaline compound is an alkali metal compound.
9. Process according to claim 8, wherein distilling the mixture is effected with such an amount of alkali metal compound that the concentration of alkali  
35 metal in the bottom of the distillation column is higher than 2 weight ppm and lower than 50 weight ppm.

9572WO

22 December 2004

Enclosure 1.2

AMENDED SET OF CLAIMS (continued)

10. Process according to any one of claim 1 to 9, wherein said alkaline compound is an alkali metal hydroxide, alkali metal carbonate or alkali metal alkoxide.
- 5 11. Process according to any one of claim 1 to 10, wherein said alkaline compound is sodium hydroxide or potassium hydroxide.
12. Process according to any one of claim 1 to 11, wherein the process further comprises feeding said bottom product to a distillation column in which cyclohexanone is distilled off as a top product.
- 10 13. Process according to any one of claim 1 to 12, wherein preparing of the mixture involves oxidizing cyclohexane in the liquid phase with an oxygen containing gas in the absence of an oxidation catalyst resulting in an oxidation mixture comprising cyclohexane, cyclohexyl hydroperoxide, cyclohexanone and cyclohexanol; treating the oxidation mixture with a cyclohexyl hydroperoxide decomposing metal salt in the presence of an alkali metal hydroxide such as to effect decomposition of the cyclohexyl hydroperoxide into cyclohexanone and cyclohexanol to obtain the mixture subjected to said distilling in the presence of an alkaline compound.
- 15 14. Process according to claim 13, wherein said preparing of the mixture further involves separating cyclohexane prior to said distilling.
- 20 15. Process according to any one of claims 1-14, wherein the process comprising oxidizing cyclohexane in the liquid phase with an oxygen containing gas resulting in an oxidation mixture comprising cyclohexane, cyclohexyl hydroperoxide, cyclohexanone and cyclohexanol; treating the oxidation mixture with a cyclohexyl hydroperoxide decomposing metal salt in the presence of an alkali metal hydroxide such as to effect decomposition of the cyclohexyl hydroperoxide into cyclohexanone and cyclohexanol, resulting in a mixture comprising cyclohexanone, cyclohexanol and cyclohexane;
- 25 separating, by distillation, cyclohexane from the mixture comprising comprising cyclohexanone, cyclohexanol and cyclohexane; separating, by distillation, low boiling compounds from the resulting mixture to obtain a top product comprising low boiling compounds and a bottom product comprising cyclohexanone, cyclohexanol and high boiling compounds;
- 30

9572WO22 December 2004Enclosure 1.3AMENDED SET OF CLAIMS (continued)

- wherein the distillation to separate cyclohexane or the distillation to separate low boiling compounds is carried out according to any one of claims 1 to 15.
- 5    16.    Process according to claim 15, wherein the process further comprises feeding said bottom product to a distillation column in which cyclohexanone is distilled off as a top product.



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